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Enhanced performance of sulfone-based electrolytes at lithium ion battery electrodes, including the $LiNi_{0.5}Mn_{1.5}O_4$ high voltage cathode

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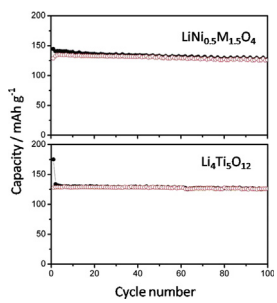
HIGHLIGHTS

- 99% coulomb efficiency at $LiNi_{0.5}M_{1.5}O_4$ cathode with sulfone–carbonate electrolyte.
- This electrolyte also yields 97% capacity retention at $LiNi_{0.5}M_{1.5}O_4$ over 100 cycles.
- Also reported is ideal capacity retention and efficiency at $Li_4Ti_5O_{12}$ anode.
- So $Li_4Ti_5O_{12}$/(this electrolyte)/$LiNi_{0.5}M_{1.5}O_4$ full cells offer safe, efficient, long life with reduced fire risk.

GRAPHICAL ABSTRACT



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ABSTRACT

In an extension of our previous studies of sulfone-containing electrolytes for lithium batteries, we report tests of sulfone–carbonate, and all-sulfone, electrolytes in a variety of anode and cathode half cells. While the all-sulfone electrolyte appears promising at the graphite anode in early cycles, it fails at the high voltage cathode and will not be further tested. On the other hand a sulfone–carbonate mixed solvent performs very well (97% capacity retention after 100 cycles with 99–100% coulomb efficiency) at the $LiNi_{0.5}M_{1.5}O_4$ (LNMO) cathode, and also at the $Li_4Ti_5O_{12}$ (LTO) anode, both of which are being developed for high power applications.

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1. Introduction

The paramount importance of developing electrochemical means of vehicle propulsion to replace oil-based internal combustion engines has been emphasized by the many recent weather catastrophies. These are of course driven by high atmospheric water contents that increase exponentially with (atmospheric CO_2-related) ocean temperature. A variety of high energy capacity systems, like the lithium–sulfur and lithium–air systems are under development in response to this challenge, but there are major problems in the way of their application. The critical short-term needs are for improvements in the performance (safety, energy density, and power) of the standard lithium ion batteries – which approach a mature technology.

Of much interest at the moment is the possibility of developing battery systems with voltage outputs some 20% higher

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than those of the lithium ion systems that currently dominate the portable energy scene, specifically, the C/$LiCoO_2$ system, voltage output 3.5–4.0 V. Electrical "pressures" of 4.7 V, acting over long periods of time, impose great stresses on the organic solvents that have served so well for the current technology. Accordingly, other lithium-transporting systems have been under investigation. These have ranged from solid (single ion conducting crystalline and glassy) ionic electrolytes (for instance, Hayashi et al. [1]), through ambient temperature ionic liquids (for instance, Kim et al. [2]), to molecular solvents of alternate chemistries, such as the sulfones of the present and recent articles [3–10]. The development of electrolytes that withstand the high voltage cathodes, will not only enable ~5 V batteries but also will support the possibility of 3.5 V batteries with safe high power lithium titanate anodes.

Sulfones have long been known to have high resistances to both reducing and oxidizing conditions and this lead to the efforts of Xu and one of us [4] to find low melting sulfones for possible high voltage electrolyte applications. Ethylmethyl sulfone (EMS) and its eutectic with dimethyl sulfone (T_e = 25 °C) were products of this effort, and there have been fluorinated [5] and oxygenated [6,7] versions since that time. All have managed to combine advantages with one or another disadvantage (for instance, low melting point but incompatibility with graphite electrodes [7], or the opposite combination [5]).

Unfortunately, sulfone solvents generally have high viscosities, which usually implies low conductivities, and this has been a persistent problem in all of the sulfone solvent-based electrolyte studies to date. Attempts to decrease the viscosity by the same strategy used for carbonate electrolytes – namely, addition of lower-melting, less-viscous co-solvents – generates a different sort of problem that also diminishes the conductivity, namely the excess coupling of lithium ion motion to that of the anionic species (usually referred to as "incomplete dissociation" or "diminished ionicity"). This is a greater problem in the lithium ion electrolyte field than is generally realized, and indeed it provided the motivation for our previous contribution [3] in which the incomplete dissociation was quantified using a Walden plot analysis. The Walden rule has been in frequent use in this laboratory for assessing the free ion character of new ionic liquids [11–13] but had not been applied previously to the problem of dissociation in lithium salt in organic solvent electrolytes. Our analysis showed that the "standard" electrolyte used in most lithium ion cells, namely $LiPF_6$ in EC-DMC, suffered a large loss in ionicity in consequence of the choice of low viscosity co-solvent used to increase the conductivity. This is an unfortunate, but unavoidable, compromise that has to be made in order to obtain the needed power output capability for the battery.

In our earlier work [3], we followed the same strategy to greatly increase the conductivity of the ethylmethyl sulfone high voltage electrolyte, choosing the only small sulfone we could identify that was capable of acting as a solvent for lithium salts, and also had reasonable electrochemical stability. This was fluoromethyl sulfone CH_3SO_2F (FMS), commonly known as methane sulfonyl fluoride. We also measured the properties of EMS with carbonate mixtures because a synergistic effect had been observed in earlier work [5] in which the sulfone under study (trifluoropropyl-methylsulfone) was found to confer its stability on the EC and DMC that were added as cosolvents. Sulfolane as electrolyte solvent was investigated in the same way. Although the electrolytes were well characterized in this work, no tests of their compatibility with anodes, like graphite/lithium intercalates, or cathodes, like the high voltage $LiNi_{0.5}M_{1.5}O_4$ (henceforward LNMO) class of spinel cathode, were reported. We have now completed that part of the overall study and report the results herein.

To anticipate our results, we find that while the all-sulfone electrolyte gives good cycling behavior at the graphite anode, it fails at the cathode, while the mixed sulfone carbonate (EMS-DMC) electrolyte yields excellent cathode function but performs only quite poorly at the graphite anode, and would need SEI-stabilizing additives for successful applications. It will be seen, on the other hand, that the cyclability, and coulombic efficiency accessible with the EMS-DMC solvent is comparable with that obtainable with the "standard" electrolyte, thus confirming and extending the findings of Abouimrane et al. [9] for sulfolane-based electrolytes at the high voltage cathode, and the more recent related work of Demeaux et al. [10]. Other reasons for giving further favorable consideration to electrolytes of the type introduced in our earlier studies will be included in the discussion section of this paper. On the other hand, based on the results to be presented below, the study of all-sulfone systems, will now be abandoned in this laboratory.

2. Experimental section

2.1. Materials

The synthesis and physical properties of the electrolytes, 1 M $LiPF_6$ in EMS-DMC (1:1 by wt.) and EMS-FMS (1:1 by wt.), have been described in our previous report [3].

For the $LiNi_{0.5}Mn_{1.5}O_4$ cathode, preformed sheets were provided by Hydroquebec Co. under arrangement with the DOE LBL-BATT research program. They comprised [LNMO 89%, carbon black 3%, vapor grown carbon fiber (VGCF) 3% and polyvinyl difluoride (PVDF) 5% in mass].

For the anodes, graphite [CGP-G8 (93.1 wt%)/PVDF(4.9 wt %)/VGCF(2 wt%)] was also obtained for the Hydroquebec source under arrangement. For the $Li_4Ti_5O_{12}$ anode, powdered material was purchased from MTI Corporation. The LTO electrode was prepared by mixing 80% active LTO material with 10 wt% carbon black and 10 wt% PVDF in N-methylpyrrolidinone (NMP) to form a slurry that was coated on an Al foil. After coating, the electrodes were dried at 100 °C overnight, and weighed before cell assembly. All the electrode sheets were cut into disks with diameter of 1.27 cm for battery assembly (see below).

2.2. Electrolyte stability characterization

The electrochemical stabilities of the binary electrolytes were determined by cyclic voltammetry (CV) using a potentiostat/galvanostat (Princeton Applied Research, VMP2). A three-electrode cell was employed with platinum as the working and counter electrodes, and lithium metal as the reference electrode. All the scans were made at room temperature at a scan rate of 5 mV s^{-1} with the voltage range of −0.3–6.3 V.

2.3. Electrolyte–electrode compatibility studies

The compatibility of the electrolytes with cathode and anodes was evaluated by using lithium-ion half-cells. Coin-type 2032 cells (20 mm diameter, 3.2 mm thickness) were assembled in an argon glove box with lithium as the counter electrode. Coin cell kits were purchased from MTI Corporation. The negative covers, spacers and springs were made of stainless steel 316 (SS-316), and the positive containers were Al-clad SS-316 to avoid the corrosion of stainless steel by electrolytes over 4.0 V. Fiber glass separators were used instead of conventional separators (polyethylene or polypropylene), which have a wettability problem in contact with sulfone molecules [7,9]. The fiber glass separators were very thick (~200 μm) compared to the 20 μm typical of the PE/PP so comparisons of rate capability with those obtained with thinner

separators are not appropriate. In spite of this, our discharge capacity was not affected by C-rates until above C/2. Charge/discharge tests were performed using a LAND CT2001A battery testing system, using groups of 3 or 4 nominally identical coin cells to check for consistency.

3. Results

In Fig. 1 we examine the cyclic voltammograms (5 mV s^{-1}) on Pt working electrodes investigated with the alternative electrolytes, 1 M $LiPF_6$ in EMS-DMC (1:1 by wt.) and 1 M $LiPF_6$ in EMS-FMS (1:1 by wt.) scanning up to 6.3 V in order to obtain the characteristic electrochemical "windows" for the two electrolytes. On the cathodic side of the window, reversible deposition and stripping of lithium is evident at 0.0 V. On the anodic side it is seen immediately that, not-withstanding the limited stability of pure DMC (~ 5.0 V on Pt [14] and only 4.2 V on a Lithium manganate cathode [5]), the EMS-DMC solvent is electrochemically stable to the same very high limit (>5.9 V vs Li^+/Li by a very conservative definition) previously reported for EMS itself on Pt [4]. The explanation for this felicitously synergistic effect [5] has been provided by the recent simulations of Xing et al. [15] which showed that the less polar carbonate was excluded from the immediate contact layer at the cathode surface by the more polar (and stable) sulfone.

In Fig. 2 we show the performance of the LNMO cathode with 1 M $LiPF_6$ in EMS-DMC electrolyte in a lithium half cell. The charge/discharge curves (Fig. 2a) confirm again that the LNMO electrode is characterized by two distinct plateaus, corresponding to the two redox processes of nickel, that is Ni^{2+}/Ni^{3+} and Ni^{3+}/Ni^{4+}, at about 4.6 V and 4.7 V versus Li, respectively. The two steps are seen most clearly in the derivative plots, dQ/dV, given in Fig. 2b. Working with this electrolyte, the LNMO delivers an initial charge capacity of 144.7 mAh g^{-1}, and the reversible discharge capacity is 128.9 mAh g^{-1}, corresponding to an initial coulombic efficiency of 89.1% comparable to that reported for all carbonate electrolytes by Xu et al. [14] After 100 cycles, our half cell retains a high discharge capacity, 126.0 mAh g^{-1}, having lost only 2.9 mAh g^{-1} capacity (Fig. 2c) *(see note added in proof)*. Furthermore, the coulombic efficiency increases with increasing cycling reaching 99.0% after some 30 cycles (Fig. 2d), thus confirming that our sulfone-based electrolyte has a good compatibility with the high voltage cathode. Comparisons with other studies will be left to the discussion section.

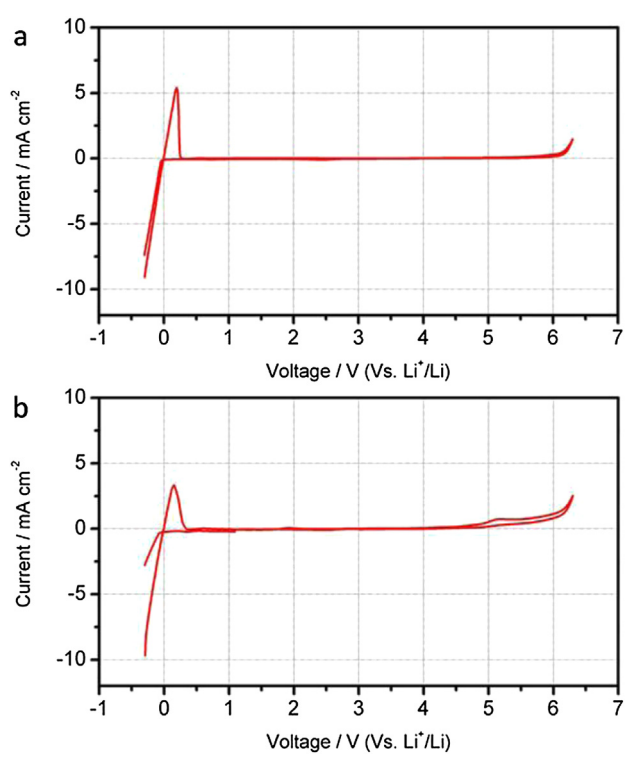

Fig. 1. Cyclic voltammograms of 1 M $LiPF_6$ in different solutions for solvent electrochemical stability characterization. (a) EMS-DMC (1:1 by wt.) and (b) EMS-FMS (1:1 by wt.). Scan rate: 5 mV s^{-1}; Pt as the working and counter electrodes and Li as the reference electrode. The scale used to accommodate the anode action conceals a small oxidative current starting at about 5.9 V, in panel (a).

Turning to the all-sulfone (EMS-FMS) electrolyte, we find (Fig. 3) that the LNMO half cell shows a reasonable charge/discharge behavior in the first cycle but, as might be expected from the CV in Fig. 1b, severe capacity decay and low coulombic efficiency are encountered above 4.5 V due to electrolyte decomposition.

In a successful battery, the electrolyte (both solvents and salts) must cope with challenges from both the cathode and the anode. For anode tests, we examined first the familiar graphite case, the dominant anode material in the lithium ion battery market, which supports lithium intercalation well when a suitable solid electrolyte interphase SEI is formed. Unfortunately, as shown in Fig. 4a, and as anticipated from previous work with sulfone electrolytes, the graphite anode shows rapid capacity fade for the EMS-DMC electrolyte half cell. Even with the carbonate co-solvent a competent SEI is evidently unable to form. We have shown that this problem with sulfone solvents can be alleviated to an extent by judicious use of SEI-forming additives such as VC [6], but this corrective has not been pursued in the present work. At the graphite electrode, the EMS-FMS-based electrolyte actually shows better performance than EMS-DMC (Fig. 4b), probably due to the fact that FMS has the same $[FSO_2]^-$ moiety as the $[FSA]^-$ anion of the lithium bis-fluorosulfonylamide salt LiFSA, which is known for its SEI-forming propensity at the graphite electrode [16]. The voltage plateau at 1.0 V in the first charge probably relates to an SEI formation process and could be used as a guide in a study of additive effects in future work.

From Fig. 4, it is clear that, without introduction of effective additives, we cannot combine LNMO cathode with a graphite anode, to take full advantage of the high voltage capability of the cathode, using our EMS-DMC-based electrolyte. However, even with good SEI formation there are safety issues with graphite anodes working with carbonate-containing electrolytes. Fortunately there is available an alternative anode, possessing kinetics as favorable as those of the LNMO cathode and combining them with greatly enhanced safety factors. This is the spinel lithium titanate that has been extensively studied for its great promise for large-scale lithium-ion batteries. Enthusiasm for this anode stems from its flat potential around 1.5 V (vs. Li^+/Li) during charge and discharge, excellent cycle life due to the negligible volume change of charging, and high thermal stability. It is especially favored for elevated temperature applications [17–22]. Furthermore, spinel LTO has no solid–electrolyte interface, which allows the electrode to meet the high power and abuse tolerance requirements needed for HEVs. Evidently then, new electrolytes without EC might work well with LTO.

Fig. 5 shows the performance of the LTO anode with 1 M $LiPF_6$ in EMS-DMC (1:1 by wt.) electrolyte in a lithium half cell. Fig. 5a and b exhibits a typical lithium storage behavior of LTO, indicating the EMS-DMC electrolyte is compatible. Not only is it compatible, but the capacity retention is 100% (Fig. 5c), and the coulombic efficiency is remarkably high (except for the first cycle) (Fig. 5d). The first cycle capacity loss might originate in our electrode preparation procedure rather than in the electrolyte. We find that the all-

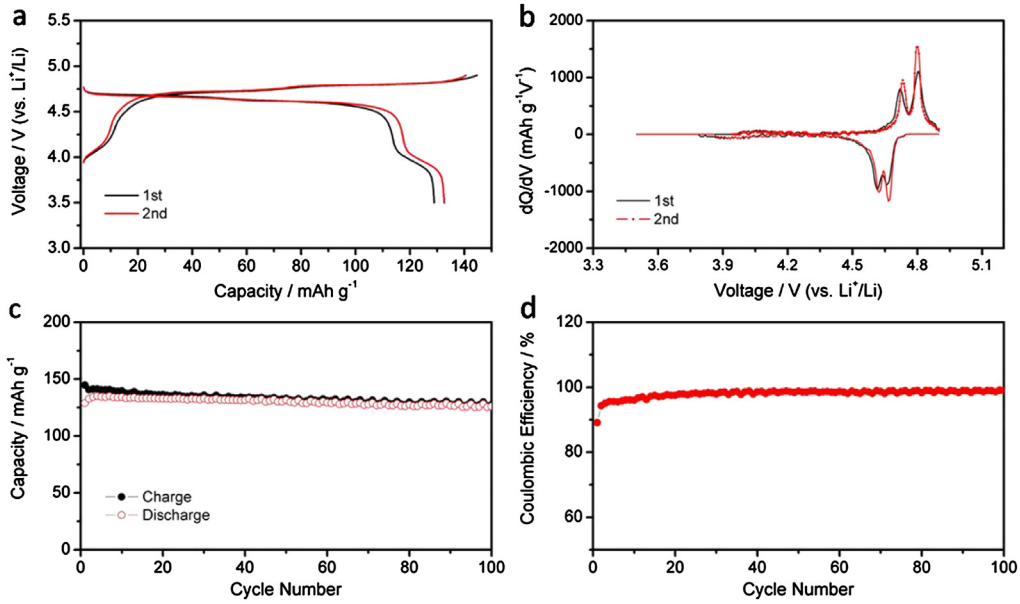

Fig. 2. Performance of the $LiNi_{0.5}Mn_{1.5}O_4$ cathode with 1 M $LiPF_6$ in EMS-DMC (1:1 by wt.) electrolyte in a lithium half-cell. Galvanostatic charge/discharge tests performed between 3.5 and 4.9 V at 0.2 C. (a) Voltage profiles of LNMO electrode cycled at 0.2 C. Note two distinct discharge plateaus for Ni^{2+}/Ni^{3+} and Ni^{3+}/Ni^{4+}, at about 4.6 V and 4.7 V, versus Li, respectively. (b) Derivative plots, *dQ/dV*, refining the successive discharge voltage steps. (c) Display of the excellent reversible cycling performance and (d) evidence for high coulombic efficiency after 30 cycles.

sulfone electrolyte EMS-FMS also fails in the LTO half cell, apparently due to a side reaction at 1.3–1.4 V. No data are reported for this case.

4. Discussion

In this work we have shown how the synergistic combination of viscous but oxidatively stable sulfone (EMS), with an acyclic carbonate that is fluid but relatively unstable (oxidatively) (DMC), can provide an electrolyte solvent with superior performance at the exciting but problematic high voltage LNMO spinel cathode. This cathode is characterized by exceptionally favorable ion exchange kinetics. The kinetics of the LNMO cathode depend on a complex management of different structural/redox effects during preparation, which affect both cathode voltage and capacity. The structural effect involves control by annealing of a thermally arrested cation site order-disorder transition [23] while the redox effect depends on control of the oxygen pressure-dependent Mn^{3+}/Mn^{4+} ratio. These effects have been explored to date on a largely empirical basis: the high capacity relative to that of other studies seen in Fig. 6 suggests that these different effects have been well balanced in the Hydroquebec cathode preparation (see Materials section).

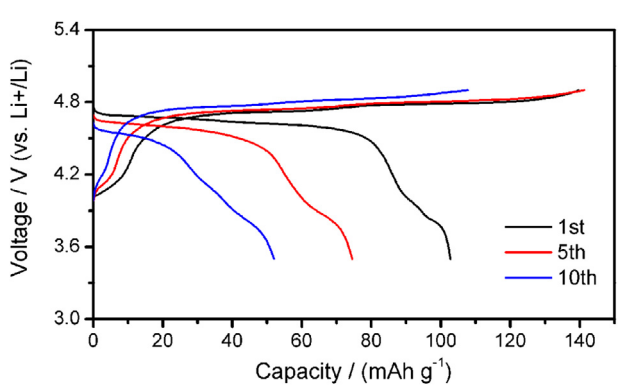

Fig. 3. Performance of the $LiNi_{0.5}Mn_{1.5}O_4$ cathode with 1 M $LiPF_6$ in EMS-FMS (1:1 by wt.) electrolyte in a lithium half-cell. The capacity decay is associated with FMS instability above 4.5 V seen in Fig. 1b CV.

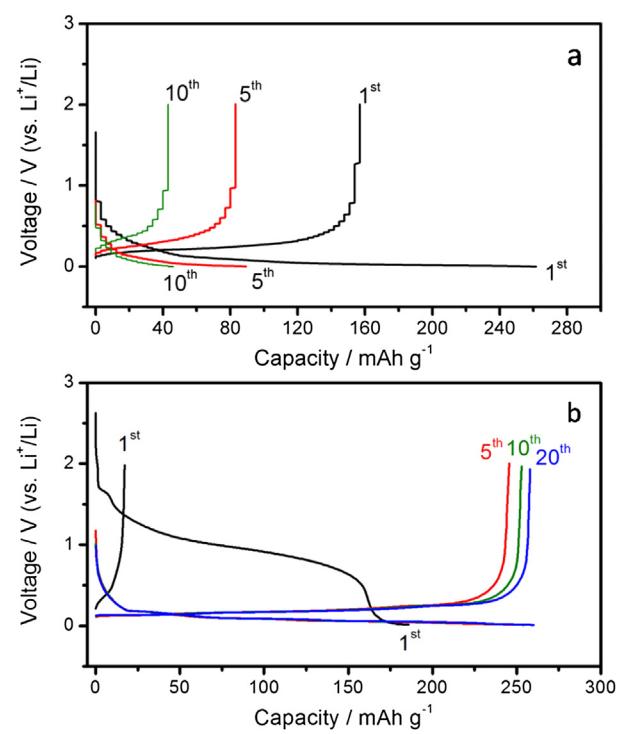

Fig. 4. (a) Performance at the graphite electrode of 1 M $LiPF_6$ in EMS-DMC (1:1 by wt.) electrolyte in a lithium half cell. The capacity fades fast. EMS-DMC can't form the protective SEI film on the surface of graphite that prevents further reaction, resulting in continuous electrolyte reduction and self-discharge. (b) Performance at the graphite anode of 1 M $LiPF_6$ in EMS-FMS (1:1 by wt.) electrolyte in a lithium half-cell. It shows much better cycling performance than EMS-DMC, though there is a large loss in the first cycle, presumably as the SEI is built up.

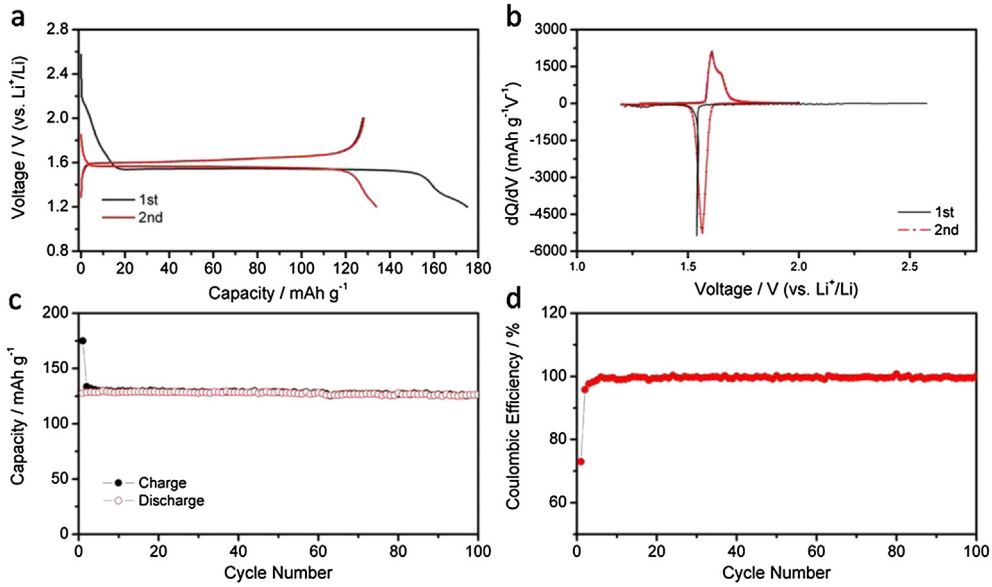

Fig. 5. Performance of the LTO anode with 1 M $LiPF_6$ in EMS-DMC (1:1 by wt.) electrolyte in a lithium half cell. (a) Illustrates the voltage profiles of the LTO electrode cycled at 0.2 C, and (b) shows the corresponding derivative *dQ/dV* response. The excellent cycling stability (c) and high coulombic efficiency (d) is especially notable.

We will not enter into the details of the cathode complexity here, but note that the effect, on cathode capacity retention (our main concern), of the annealing temperature after cathode formation, was examined in the early and detailed paper of Takahashi et al. [24]. The annealing temperature was found to affect the capacity but not the capacity retention, at least in their lithium half-cell study employing the standard EC-DMC-based electrolyte.

It is useful to compare the Takahashi findings for different cathode annealing temperatures, with those of the present study, and also with those of the more recent study by von Croce et al. [25]. In the latter study, which also involved an all-carbonate electrolyte formulation (given in figure legend), the capacity retention was relatively poor in the initial preparation but was greatly improved by inclusion in the electrolyte of an additive that was originally intended to control the electrolyte flammability. The comparison is made in Fig. 6. Of course, with sulfones there is no need to introduce antiflammability additives, because numerous studies testify to their low fire hazard advantage [9,26–28].

While there have, of course, been a large number of recent reports on cell performance with LNMO cathodes, the only case

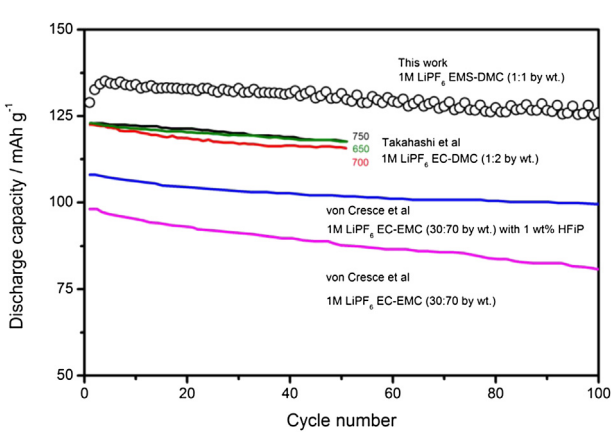

Fig. 6. Comparison of capacity retention in the present half cell with LNMO, with that found in three of the earlier all-carbonate electrolyte studies, including a stabilizing additive (see text for details).

demonstrating a capacity retention superior to that of the present work has been the recent study of Zhang et al. [29]. These authors used a new series of fluorinated electrolytes, and obtained excellent capacity retention even at 55 °C [29]. While the capacity retention of Ref. [29] is indeed better than that of the present work, the fact that the new electrolyte involves fluorination implies there will be a significant cost disadvantage. (Their capacity retention data were reported for a full cell and therefore are not strictly comparable to the half-cell studies usually reported.) Furthermore, the conductivities of all three of the electrolytes used to achieve the high capacity retentions of Ref. [29], prove to be lower that of our formulation [3] according to the SI of Ref. [29]. As mentioned also in the experimental section, our half-cell capacities are unaffected by discharge rates up to C/2, even in the unfavorable button cell configuration. Small amounts of the electrolyte of Ref. [29] might well serve as a favorable additive to improve our system performance further, although tests of additives like HFiP [25], LiBOB [30,31], and others to be described in a separate publication, have shown only very modest improvements.

The excellent cycling performance and coulombic efficiency, shown in Fig. 5c and d, respectively, make the LTO anode highly suitable for matching with the high voltage LNMO cathode when using our electrolyte. Such compatibility was already well demonstrated in Ref. [29], and has now been extended in some detailed contact stability studies made recently by Demeaux et al. [10]. These authors report that EMS-DMC gives the best stability in long term contact with anode and cathode. Since the conductivities of the sulfone carbonate electrolytes can now be raised to more than half of the standard electrolyte value by use of extra components, the combination LTO/$LiPF_6$-in-EMS-DMC/LNMO cells can therefore be expected to lend itself to fabrication of high power batteries characterized by long cycle life, high power density and modest cost.

5. Concluding remarks

Considering the benign character of EMS (its close relative dimethyl sulfone is even a health food ingredient) and the lowered fire risk (both low flammability of sulfones, and absence of

thermal runaways [9,26,27], there would seem to be much reason for further study of the present type of system. Safety tests have not been included in our study but their consideration may help establish electrolytes of the present genre as a leading choice for vehicle propulsion systems and other heavy duty applications.

The results reported here are promising but surely not optimal. For instance, for simplicity we have limited the electrolytes of this study to those containing only two components, but the most favorable sulfone from the point of view of low temperature stability is a eutectic mixture of EMS with its smaller, but higher melting, symmetrical relative, dimethyl sulfone ($T_{eut} = 25$ °C [4]). Indeed this modification of the present electrolyte has very favorable characteristics, on which we will report separately (Lee, Xue and Angell, in preparation).

Note added in proof

In a very recent paper (S.R. Li, C.H. Chen and J.R. Dahn, JECS 160, A21666–A2175 (2013)) the importance of time, not cycle number, is emphasized for the presentation of cell performance because of the importance of time-dependent parasitic reactions such as electrolyte oxidation and SEI growth. We therefore add here that the data presented in our ***Figure 2*** were acquired over 900 hr of continuous cycling. Furthermore, in related work with half cells similar to those of the present paper that had been cycled 200 × (1800 hr), we have resumed cycling after a two months hiatus, and have found the cell performance unchanged except for the initial cycle which, like the original opening cycle, had a lower coulombic efficiency (details being reported separately)

Acknowledgments

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